## Resources of UNIT OPERATIONS & SEPARATION PROCESS Lab (CHE 327)

### 1) Molecular diffusion of gases

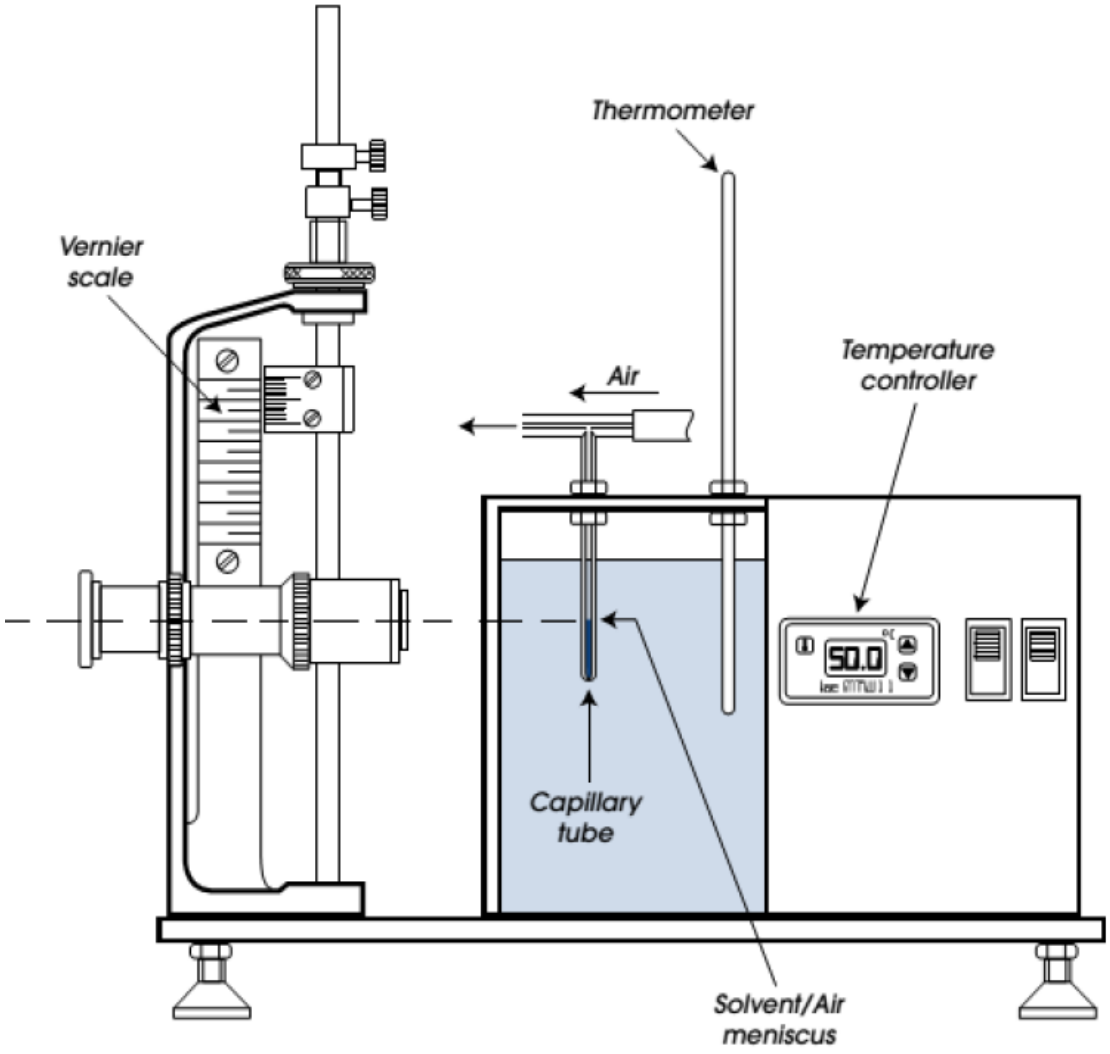



**Fig 1: Molecular diffusion of gaseous**

## Technical description

The liquid that is to be volatilized is placed in capillary tube. The capillary tube is placed inside a water bath whose temperature is carefully controlled by a controller. The height of the liquid that decreases by time is observed using microscope equipped with Vernier height gauge.

### 2) Convection Drying

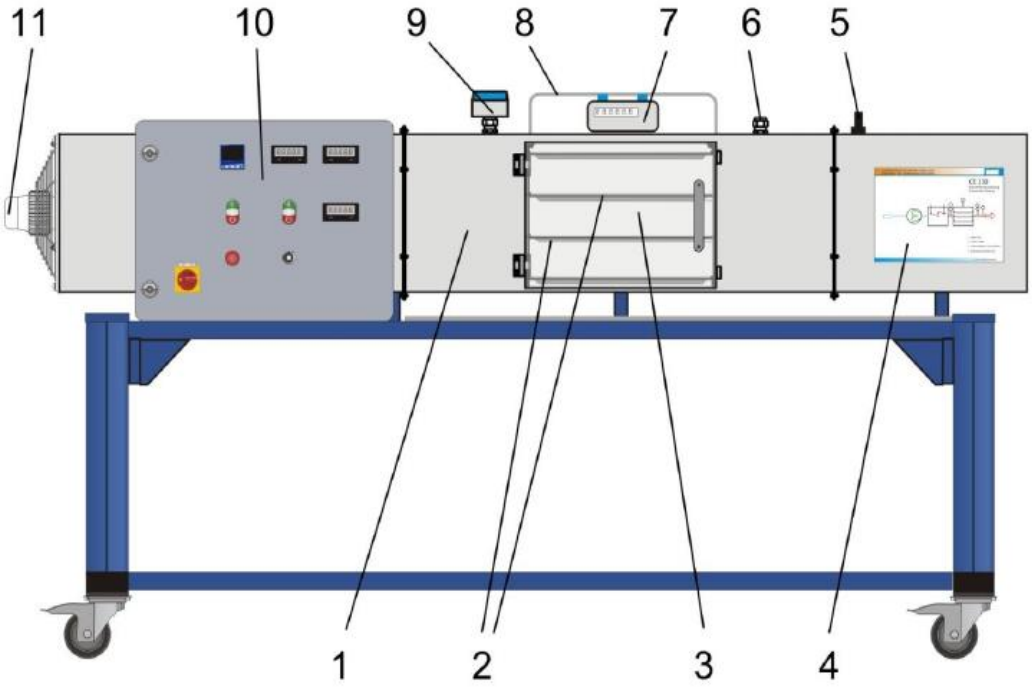



**Fig 2: Convection drying unit**

**Description**

In many cases, drying is carried out using warm air at atmospheric pressure. This drying process is called air drying. If drying is done at significant sub atmospheric pressure, it is called vacuum drying. In some cases, the moisture in the material is cooled and frozen and using convection, conduction or radiation heat transfer, and the freezing moisture can be sublimed. This method of drying is known as freezing drying. One of the most important analyses in drying is rate of drying curves. The equilibrium moisture contents of various materials cannot be predicted and must be determined experimentally. Similarly, since the basic mechanism of rates of drying is quite incomplete thus it is necessary in most cases to obtain some experimental measurements of drying rates.

1. Drying channel
2. Drying plates
3. Transparent door
4. Process schematic
5. Air velocity sensor

6. Measuring point for humidity and temperature

7. Digital balance

8. Bracket for frying plates

9. Measuring point with humidity and temperature sensor

10. Switch cabinet with digital displays

11. Fan

**3) Wet cooling tower**

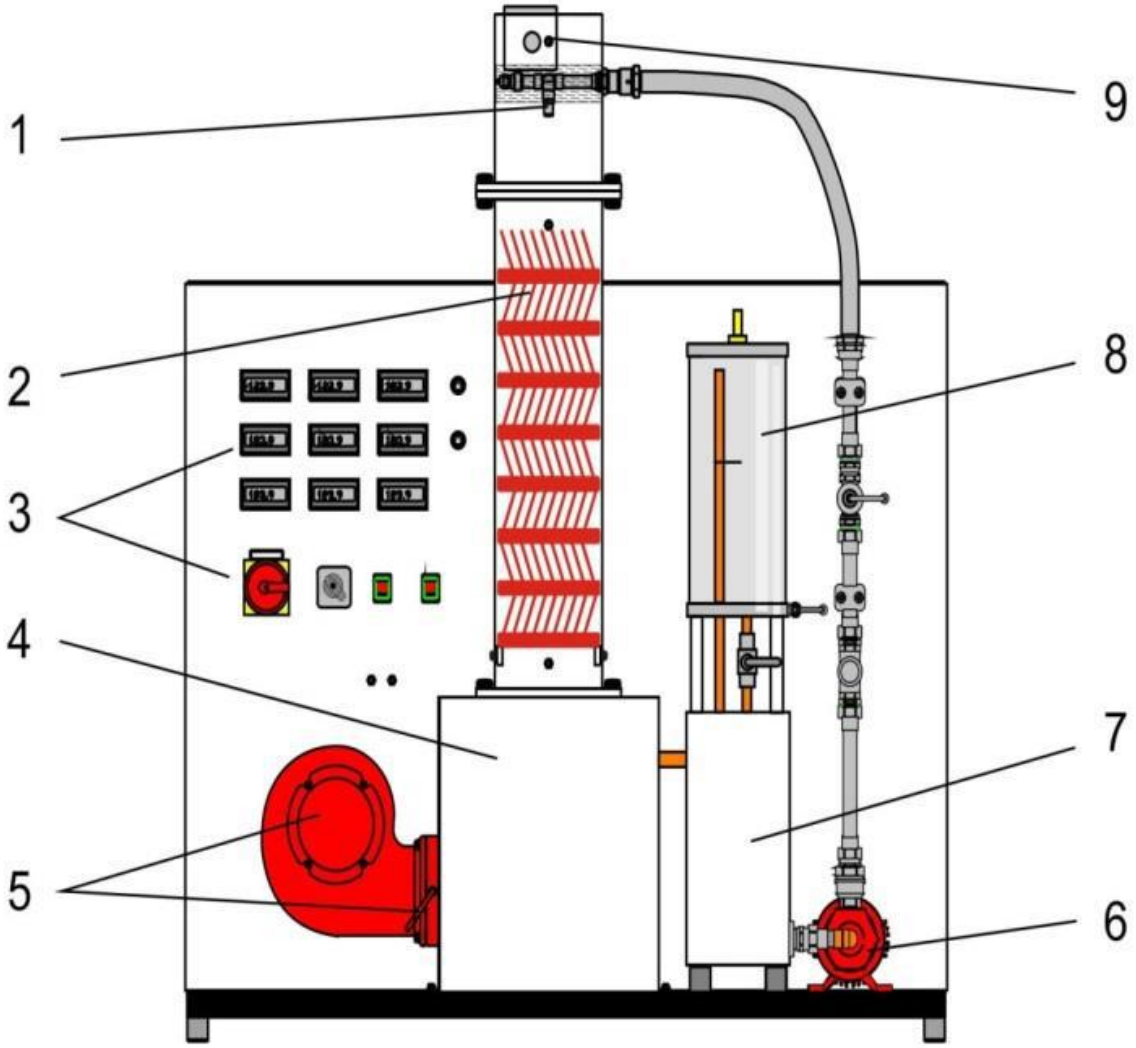


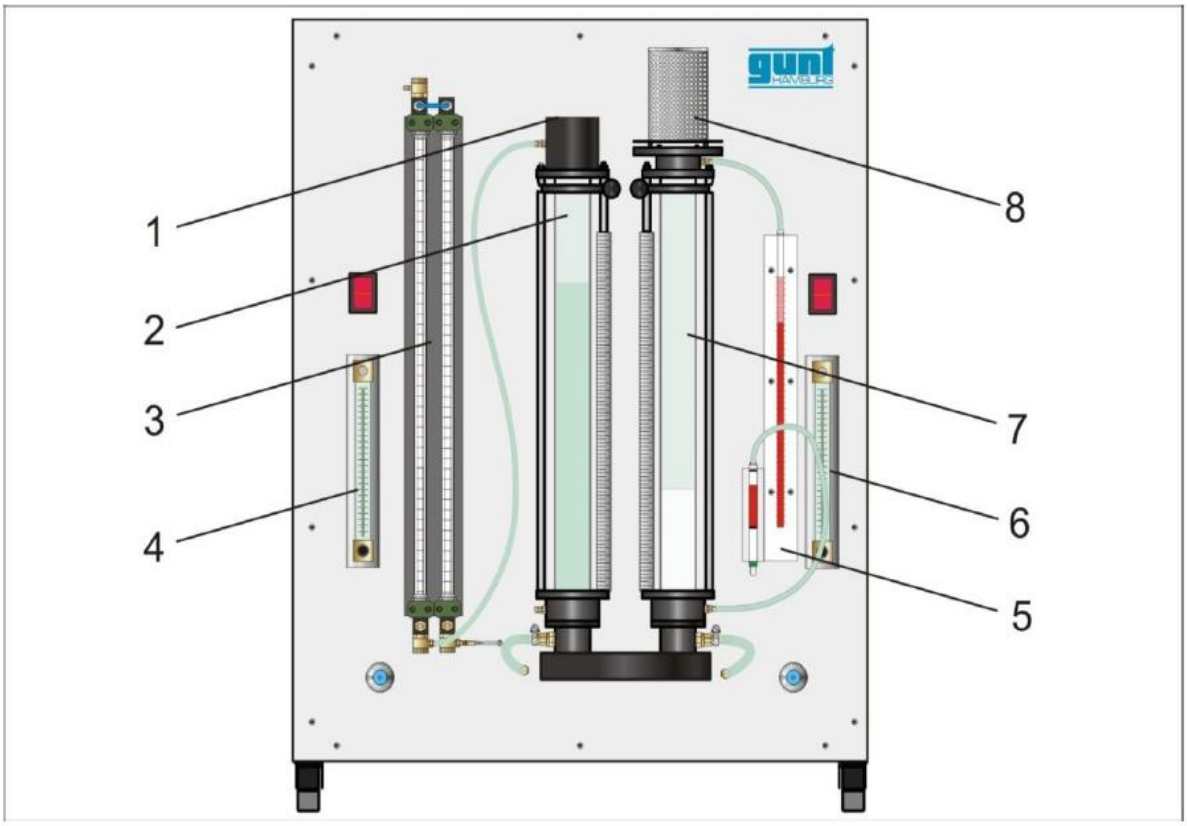

**Fig 3: Wet cooling tower**

**Technical description**

The main wet cooling tower unit can be seen in figure 1. The water is heated using heating element in a tank (7), pumped (6) and then sprayed from the top of the column through nozzle (1) and intimately contact with cooling air due to the packing (2). The air comes in from the air chamber (4) at the bottom of the

column. The air flow rate is adjustable using fan (5). Due to evaporation during the course of the process, the makeup water (8) is required to maintain enough water for the process. All parameter readings including water and air flow rate as well as temperature can be collected manually from the display and control buttons (3). The column is also equipped with temperature/humidity sensor (9) at the top and the bottom.

**4) Fluidized bed**




1. water overflow 2. test tank for water 3. 2-tube manometer 4. flow meter for water 5. U-tube manometer 6. flow meter for air 7. test tank for air 8. filter

**Fig 4: Fluidized bed apparatus**

**Technical Description**

Fluidized beds are widely used in process technology. One of important applications in chemical technology is fluidized bed reactor in which variety of multiphase chemical reactions is carried out. In a fluidized bed, a layer of fine granular solid matter is loosened by a fluid flowing through it to such extent that the particles of solid matter are free to move within certain limits. The layer of solid material takes on similar properties to a fluid. To characterize a fluidized bed, the pressure loss of the fluid flowing through

the bed can be used. When a fluid flows through the mass initially the pressure underneath the mass increases as the flow speed increases until the pressure forces match the weight of the mass, and the mass becomes suspended. With further increasing flow rate, the layer set in motion and reaches a fluidized state. At this condition the static solid like state changes to dynamic like state. The pressure loss now remains almost constant even with further increasing flow rate. From a certain flow rate the particles at the top no longer fall back into the fluidized bed, they are drawn off by the fluid flow and removed.

**6) Solid liquid extraction**


**Fig 6: Solid liquid extraction**

## Technical Description

Solid liquid extraction unit as shown in the diagram (1) has a spiral conveyor (2) to feed the extraction material into revolving extractor in which the extraction process occurs. This spiral conveyor can feed the solid up to 20 l/h with power consumption up to 4 W. Revolving extractors is driven by a motor (4) with speed up to 9 h and power consumption up to 0.9 W. The solvent is then fed using several peristaltic pumps (5) into the revolving extractor. The pump can feed the solvent at maximum volumetric flow rate of 25 l/h at 300 rpm. Solvent (liquid), extract (liquid) and raffinate (solid residue) are charged into the tanks (6). The mode of extraction can be changed by changing the selector valves (7) and the solvent can also be heated using a heater (8) with power consumption of 330 W. All reading and controls are placed in a cabinet (9).

## 7) PARTICLE SIZE ANALYSIS (SIEVING) and ANGLE OF REPOSE

**Fig 7: Sieve analysis**

**Technical Description**

Small particle is one of chemical engineering interest. An understanding of the characteristics of

masses of particulate solids is necessary in designing processes and equipment for dealing with streams containing such solids. One way to distinguish the particle size is by sieving. In each test sieve consists of a woven wire screen with square apertures of known size and is used for the measurement of particle size. The necessary vibrating motion is imparted by a mechanical shaker to ensure reproducible results in a relatively short time. Particle size may be specified by quoting the size of two screens, one through which the particles have passed and the other on which they are retained. However, sieving is usually used to measure size distribution. The results of sieve test may be presented in a variety of ways either in tabular or graphical form. The other way to learn characteristics of solid material is by measuring the angle of repose. The angle of repose is the steepest possible angle with the horizontal at which the material will stand when piled. Moisture content of the material is often a controlling factor and the percentage of fine material in the mass has a decided influence on the angle as the fine materials carry the bulk of moisture. Similarly, the angle depends upon the type of material including the shape and smoothness of the individual particles and their overall compactness.

**8) Sedimentation**

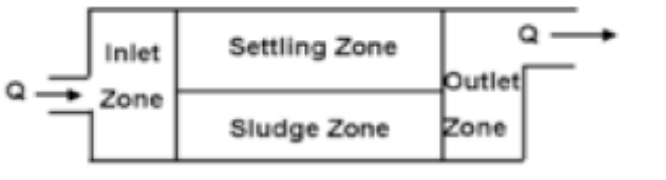



**Fig 8 (a) : The Four Functional Zone of Continuous Sedimentation Process**

**Description**

Sedimentation is a physical process of separating suspended solid in a liquid using gravity. The suspended solids can settle down if the size is greater than 10 μm. the smaller the size the more difficult for the solids to settle down because Brownian motion and electrostatic

force balances the gravitational force. Unless coagulant is added, it is not likely to settle down very small particle naturally.

In typical sedimentation tank, there will be four functional zones. Inlet zone is the zone where all solid particles are well mixed and flow in the same direction as the liquid. The settling occurs in settling zone as the liquid continue flowing to outlet zone. Most of solid particles will be settled and collected continuously in the sludge zone. In this experiment, to determine the actual sediment content at inlet and outlet to the sedimentation tank, samples of the mixtures are taken.

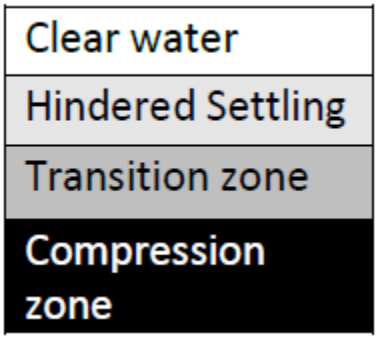
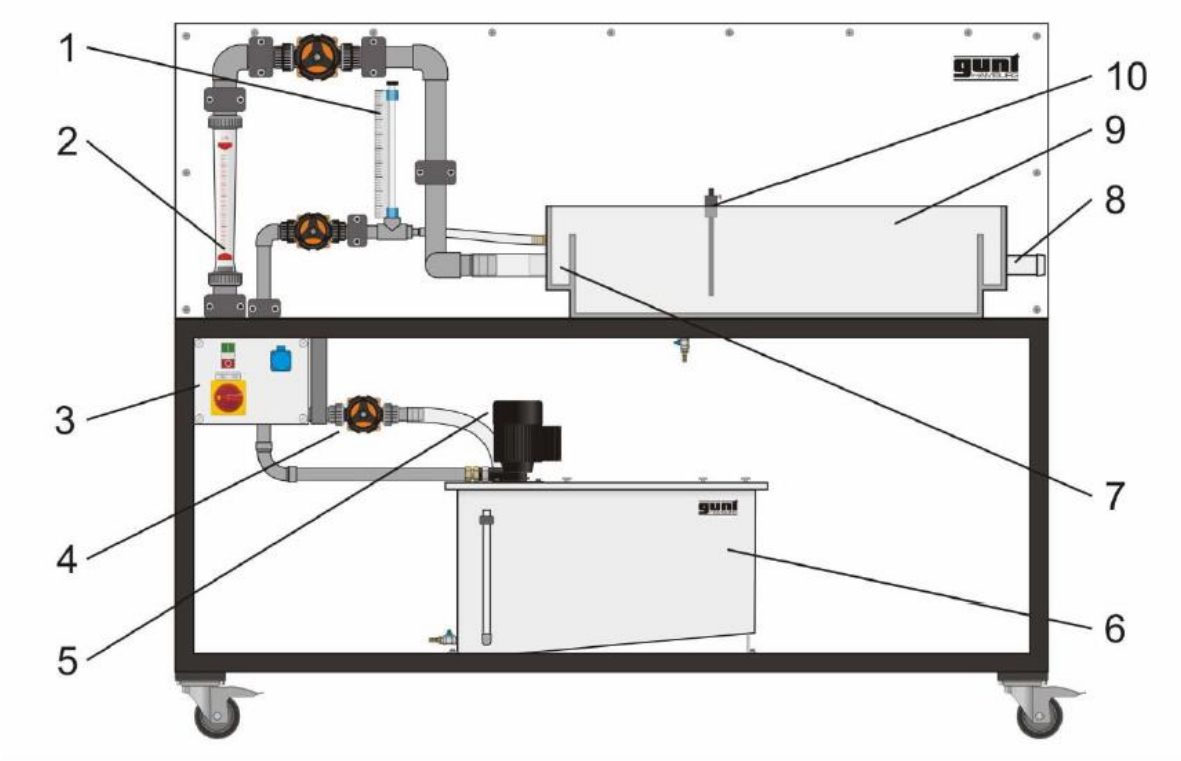



**Fig 8 (b): Four Zones of batch sedimentation**

In hindered settling, particles become quite close and the liquid is flowing upward due to displacement by settling particles. This will reduce the particle settling velocity. At the bottom of sedimentation column, the particles can contact each other and indeed the particle concentration becomes very high. The settled solids are compressed by the weight of overlying solids and water is squeezed out while the spaces between solid particles are getting smaller. This zone is known as compression zone. Between hindered settling zone and compression zone there will be transition zone.




1. Suspension flow meter
2. Fresh water flow meter
3. Switch box
4. Bypass
5. Suspension pump
6. Suspension tank
7. Fresh water/suspension mixing zone
8. Outlet
9. Sedimentation tank
10. Baffle plate

**Fig 8 ©: Sedimentation Process**

## 9) Gas Absorption

## Technical Description

Gas absorption is a unit activity where dissolvable parts of a gas blend are broken up in a fluid. Most gas absorption goes for division of acidic pollutions from blended gas streams. These acidic polluting influences incorporate carbon dioxide ($CO_2$), hydrogen sulfide ($H_2S$), sulfur dioxide ($SO_2$), and natural sulfur mixes. The most significant of these are $CO_2$ and $H_2S$, which happen at convergence of five to fifty percent. Gas ingestion at a modern scale is most normally polished in pressed towers. A pressed pinnacle is basically a bit of pipe set on its end and loaded up with dormant material or "tower pressing." Liquid filled the highest point of the pinnacle streams down through the pressing; gas siphoned into the base of the pinnacle streams counter right now upward. The private contact among gas and fluid accomplished along these lines impacts the gas ingestion. Absorption is a basic procedure in thermal process engineering. Absorption is used to separate one or more gas components from a gas flow using a solvent

(detergent). The CE 400 Gas Absorption unit provides a clear method of separating a gas mixture containing air and $CO_2$ by absorption in water. In the gas mixture section, air is mixed with $CO_2$. Mixing is carried out manually using 2 control valves and 2 rotameters. In the absorption section, the $CO_2$ is washed out of the gas mixture in a glass absorption column using water as solvent. In the desorption section, the $CO_2$ is separated from the water in a glass desorption column. The key component of the trainer is the glass packed column, in which the gas mixture is brought into contact with water. During operation, samples of the gas mixture are taken at the gas inlet, in the center of the column and at the gas outlet.

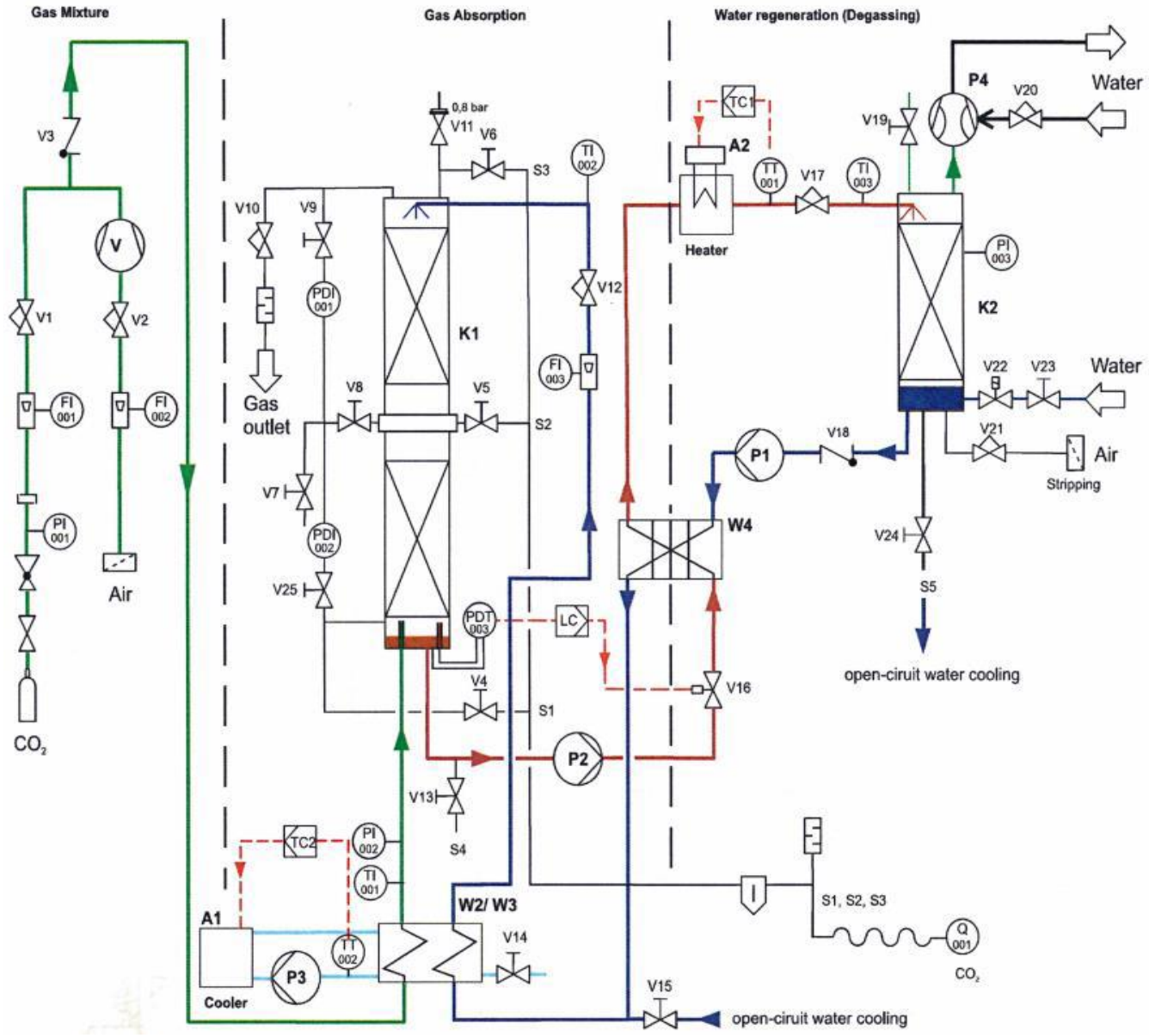



**Fig 9: Process diagram for the gas absorption system**

## 10) Distillation of binary mixtures (Ethanol and water) by Simple/batch distillation

SIMI

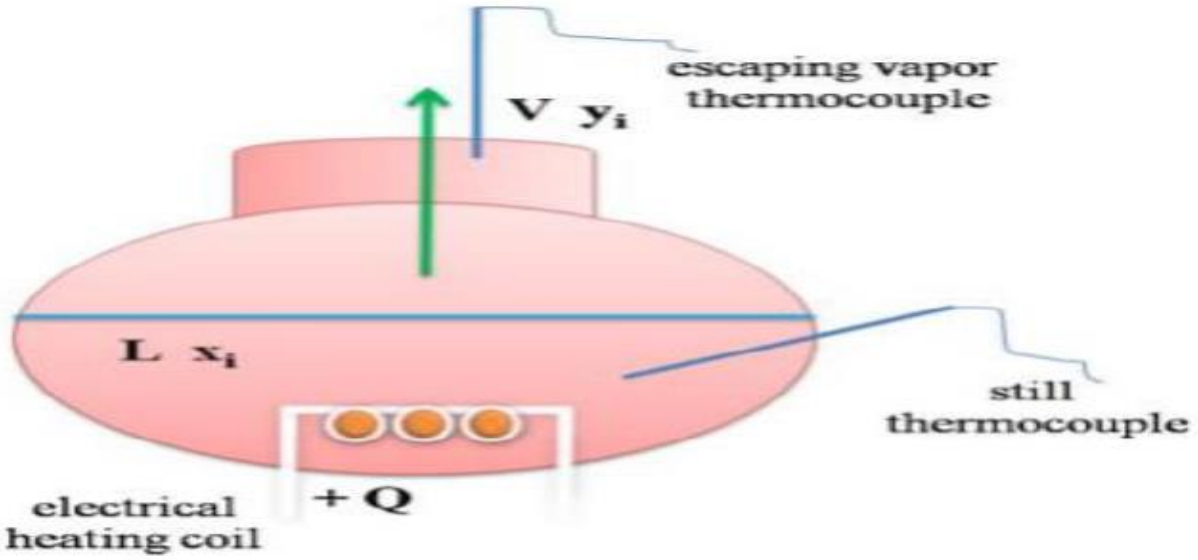



**Fig 1: Simple distillation set up**

**Fig 10 (a): Distillation of binary mixtures**

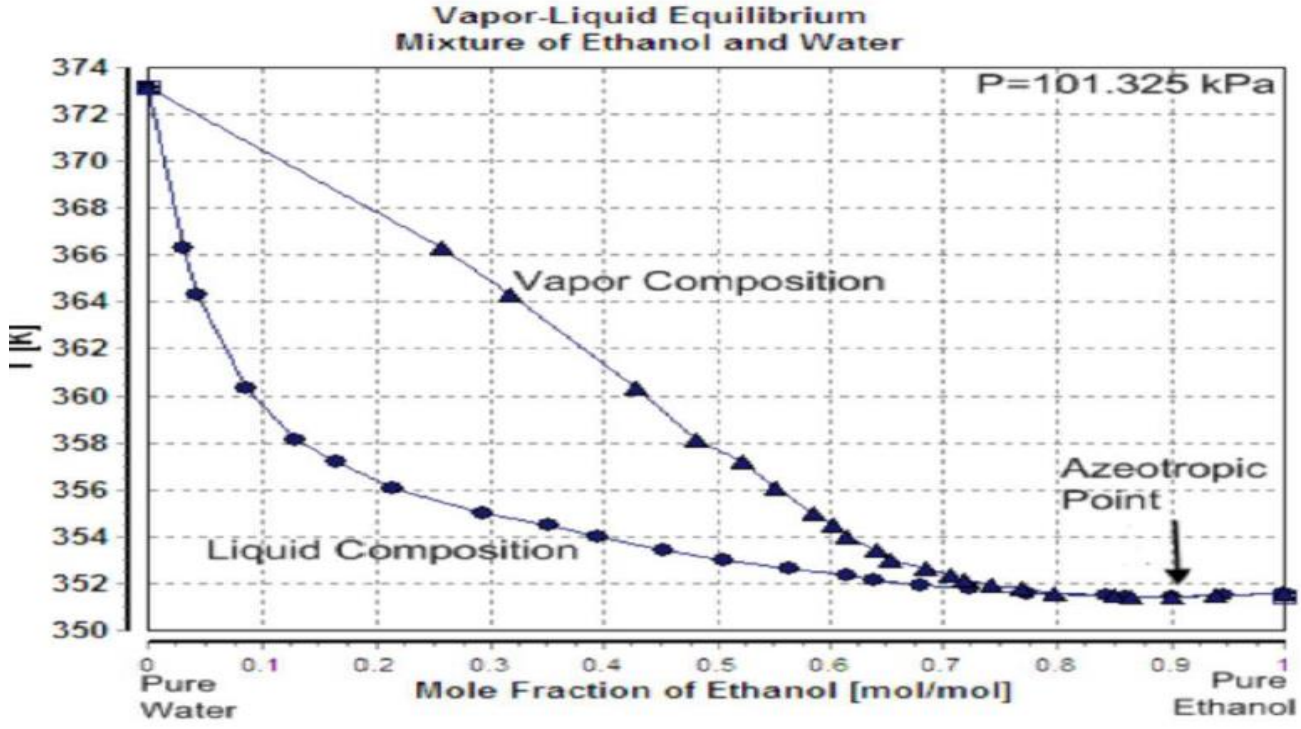



**Fig 10 (b): T-x-y diagram of ethanol water mixtures at 1 atm pressure**

The function of distillation is to separate, by vaporization, a liquid mixture of miscible and volatile substances into individual components or, in some cases, into groups of components. The separation of a mixture of alcohol and water into its components; of liquid air into nitrogen, oxygen, argon; and of crude petroleum into gasoline, kerosene, fuel oil, and lubricating stock are examples of distillation. Suppose there are two components, A and B. Both of these components are found in both phases. There are four variables: pressure, temperature, and concentrations of component A in the liquid and vapor phases (the concentrations of component B are unity less those concentrations of A). If the pressure is fixed, only one variable, e.g., liquid-phase concentration, can be changed independently and temperature and vapor-phase concentration follow. In practice, distillation may be carried out either of two principal methods. The first method is based on the production of a vapor by boiling the liquid mixture to be separated and condensing the vapors without allowing any liquid to return to the still. There is then no reflux, which is called batch distillation. [1-2]. The second method is based on the return of part of the condensate to the still under such conditions that this returning liquid is brought into intimate contact with the vapors on their way to the condenser. Either of these methods may be conducted as a continuous steady state distillation process, including single-stage partial vaporization without reflux (flash distillation) and continuous distillation with reflux (rectification), for systems containing only two components. The diagram of the system is given in **Fig 10 (a).** [3-4]. In this system it can be investigated the principles that rule the mass and energy transference, as well as determine the optimal operation point to carry out a big quantity of separations. Distillation system is basically composed by a boiler on which two types of interchangeable columns can be adapted (plate columns and Raschig Rings column), a reflux system and a tank for the distillation. · The steam that goes to the head of the column is sent to a total condenser. · The cooling water flow that crosses the condenser is regulated and indicated by the flow meter. · The pressure loss in the column can be measured with a manometer. The temperatures of the system are measured by temperature sensors placed in strategic positions.

**11) Liquid liquid extraction**

**Designation of the substances involved**

The simplest form of **liquid-liquid extraction** involves three liquids:

**Transition component** -------- Ethanol

**Carrier liquid**----------Vegetable oil

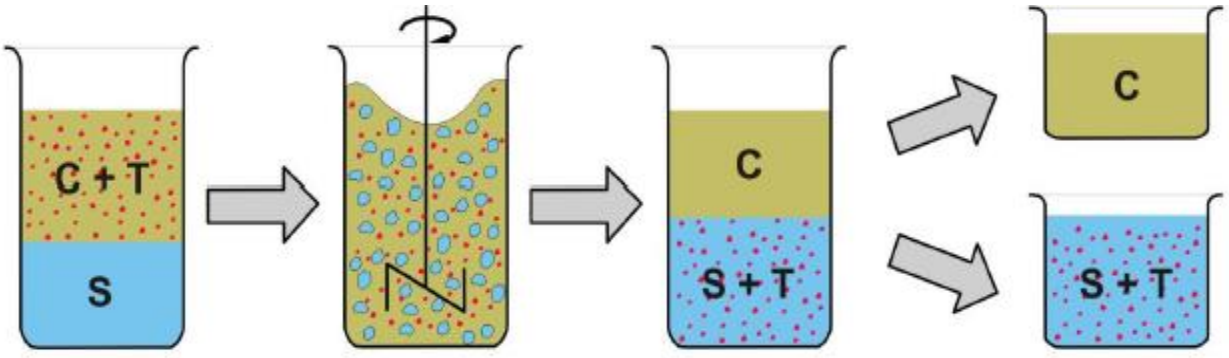
**Solvent**---------- -----Tap water

**Phenomena of Liquid-Liquid Extraction & Theory**

The liquid mixture of the transition component and carrier liquid is called the **feed**. In the feed, the **transition component** is dissolved in the **carrier liquid**. **Carrier liquid** and **solvent** together form the **sol- vent system**. The transition component must be soluble in both the carrier liquid and the solvent.

The carrier liquid and solvent should together form a **phase boundary** in order to allow the separation of the two phases. Therefore, carrier liquid and solvent should be as mutually insoluble in each other as possible. The two **phases**, which we get at the end of **liquid-liquid extraction** are called the **extract** and the **raffinate**. The **extract** is essentially a solution of the transition component in the original solvent. The **raffinate** is essentially the original feed, less the extracted proportion of the transition component.

On the left is the beaker with two liquid phases. **Carrier liquid** and **solvent** together form the **sol- vent system**. The carrier liquid and solvent together form a **phase boundary**. They are insoluble in one another. This is the condition so that separation into two phases can occur after the actual extraction. Also required is a clear density difference between the carrier liquid and solvent as shown in **fig 11**




| Liquids involved | Abbreviation |
|---|---|
| Transition component | T |
| Carrier liquid | C |
| Solvent | S |

**Fig 11: Beaker experiments with showing carrier liquid and solvent**

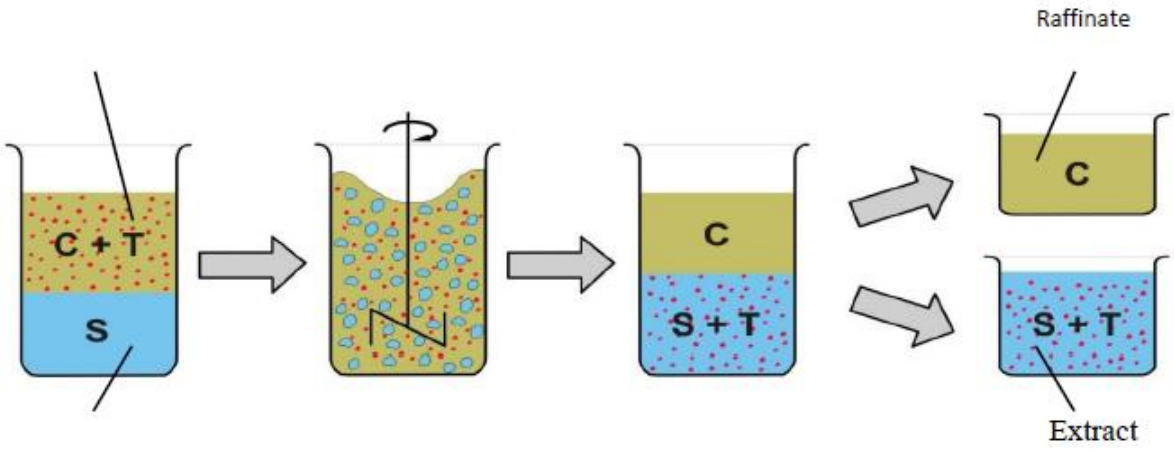



**Fig 2: Liquid-liquid extraction, identification of incoming and outgoing materials**

The following terms are common in **liquid-liquid extraction**:

The liquid mixture of the transition component and carrier liquid is called the **feed**.

The two liquid phases, which we get at the end of **liquid-liquid extraction** are called the **extract** and the **raffinate**.

The **extract** is essentially a solution of the transition component in the original solvent. The **raffinate** is essentially the original feed, less the extracted proportion of the transition component.

This results in: From solvent and feed we get raffinate and extract, as shown in **Fig 2**

The aim of liquid-liquid extraction is to transform as high a proportion of the transition component from the feed into the extract as possible.

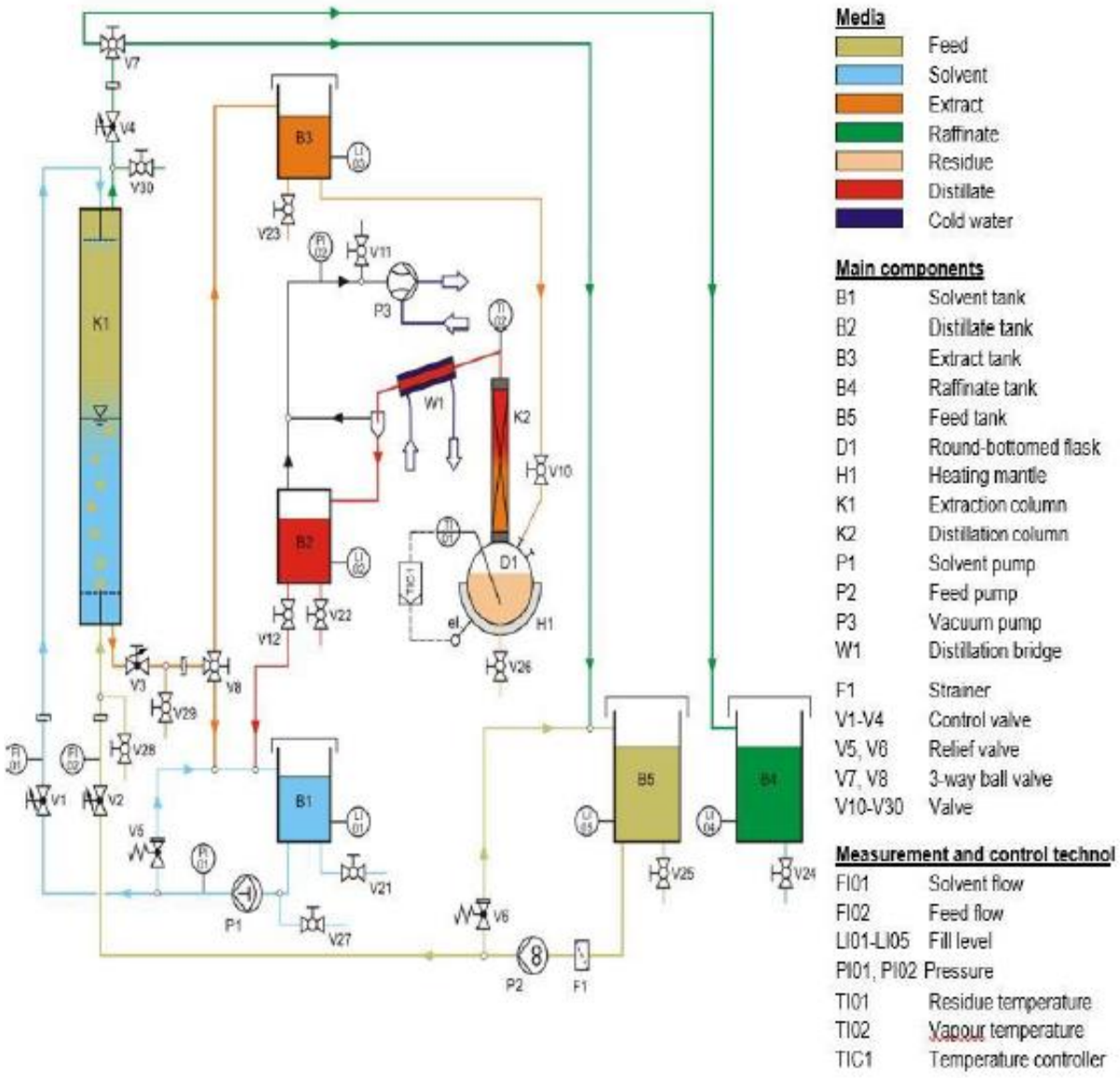


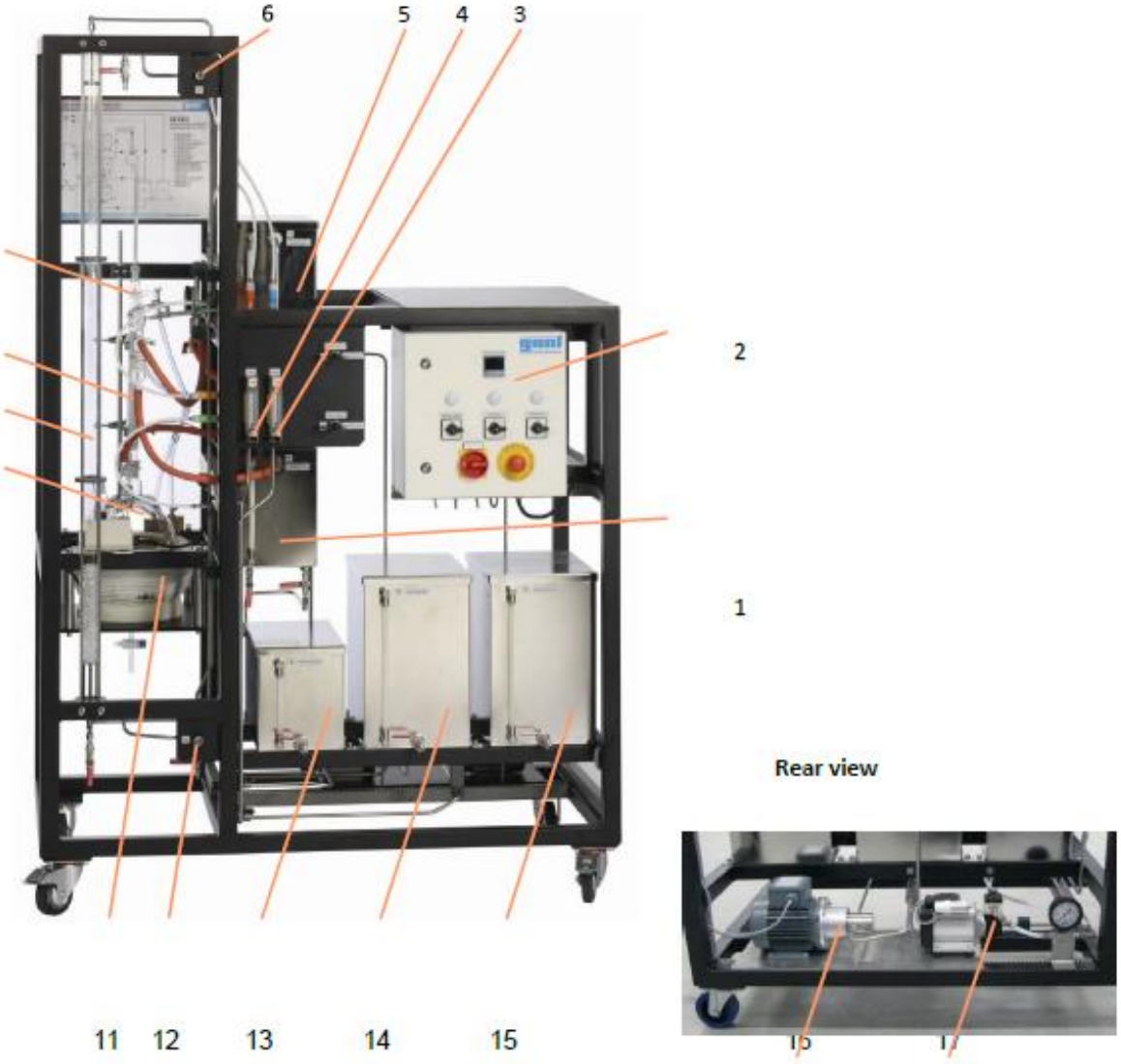

**Fig 11 (b) Process flow sheet of extraction Column**

The **fig 11 (b)** shows the process diagram for the **CE 620 liquid-liquid extraction** unit. From the top of the extraction column the feed returns to the feed tank (**feed return**). The solvent is continuously pumped from the **solvent tank** B1(13) by the **solvent pump** P1(17) into the top of the extraction column (**solvent inflow**). The solvent moves downwards in **counterflow** to the feed. From the bottom of the extrac- tion column, the solvent returns to the solvent tank (**solvent return**).

The driving force for the counterflow is the **density difference** between solvent and feed. Therefore, the material and/or the mixture with the lower density must be pumped into the bottom of the extraction column. **Fig 4** shows the front view of LLE set up.

## Device design

Front view




1 Distillate tank (B2)
2 Control cabinet
3 Regulating valve for solvent inflow (V1)
4 Regulating valve for feed inflow (V2)
5 Extract tank (B3)
6 Regulating valve for feed return (V4)
7 Distillation bridge (W1)
8 Distillation column (K2)
9 Extraction column (K1)
10 Round-bottomed flask (D1)
11 Heating mantle (H1)
12 Regulating valve for solvent return (V3)
13 Solvent tank (B1)
14 Feed tank (B5)
15 Raffinate tank (B4)
16 Feed pump (P2)
17 Solvent pump (P1)

**Fig. 4 Overview of the CE 620 liquid-liquid extraction system (Front view)**

**Fig 11 ©: Counter current extraction column**

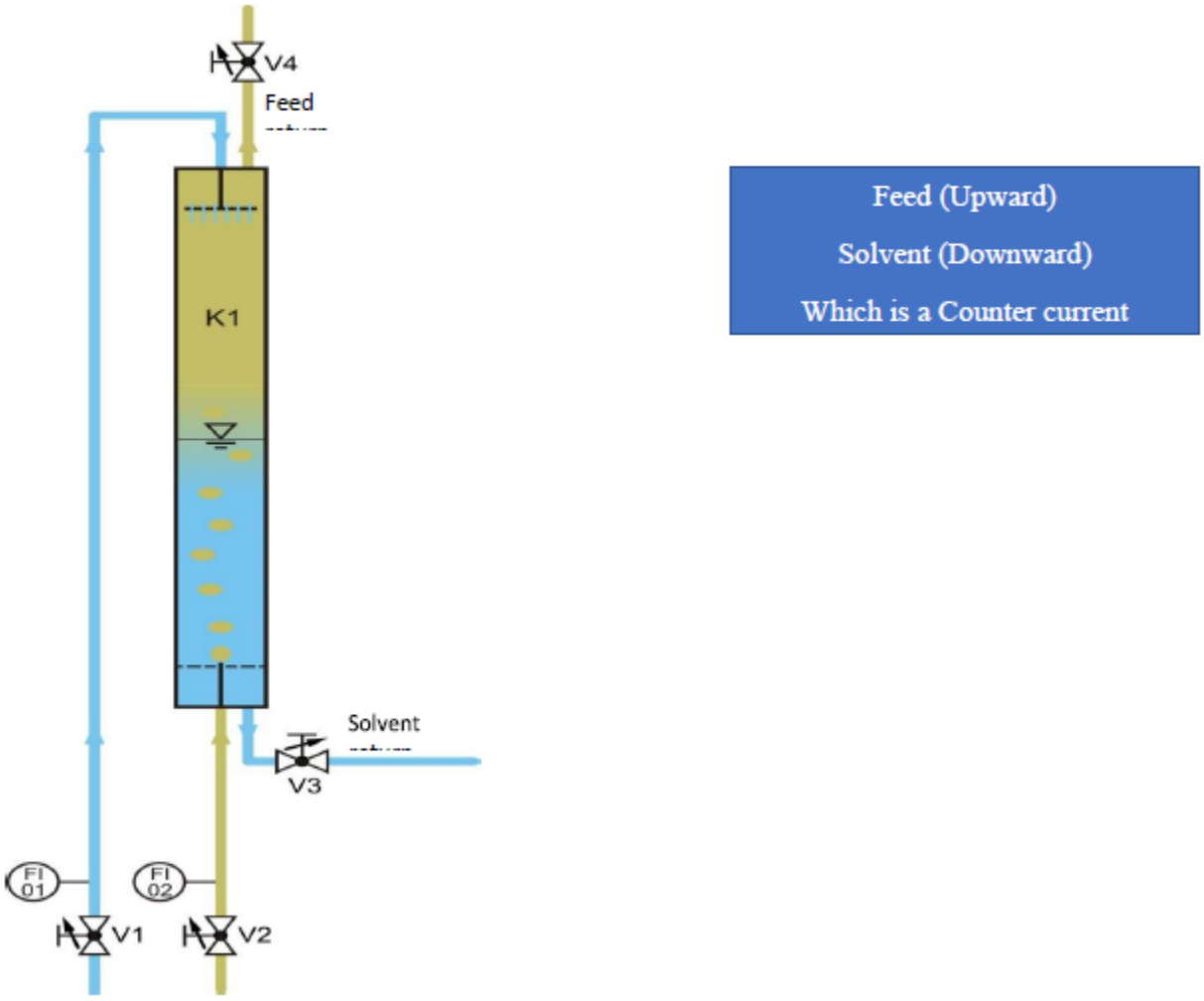



**Fig 11 ©: Counter current extraction column**

## LLE for batch mode- without distillation

The mass transfer of liquid-liquid extraction takes place in the **extraction column** K1(9). This is where **feed** and **solvent** meet. When this hap- pens, a part of the **transition component** is extracted from the feed into the solvent.

The feed is continuously pumped from the **feed tank** B5(14) by the **feed pump** P2(16) into the bottom of the extraction column (**feed inflow**). The feed moves upwards in the extraction column. **Fig 5** shows the Counter current extraction column.

## 11 (d): LLE for continuous mode

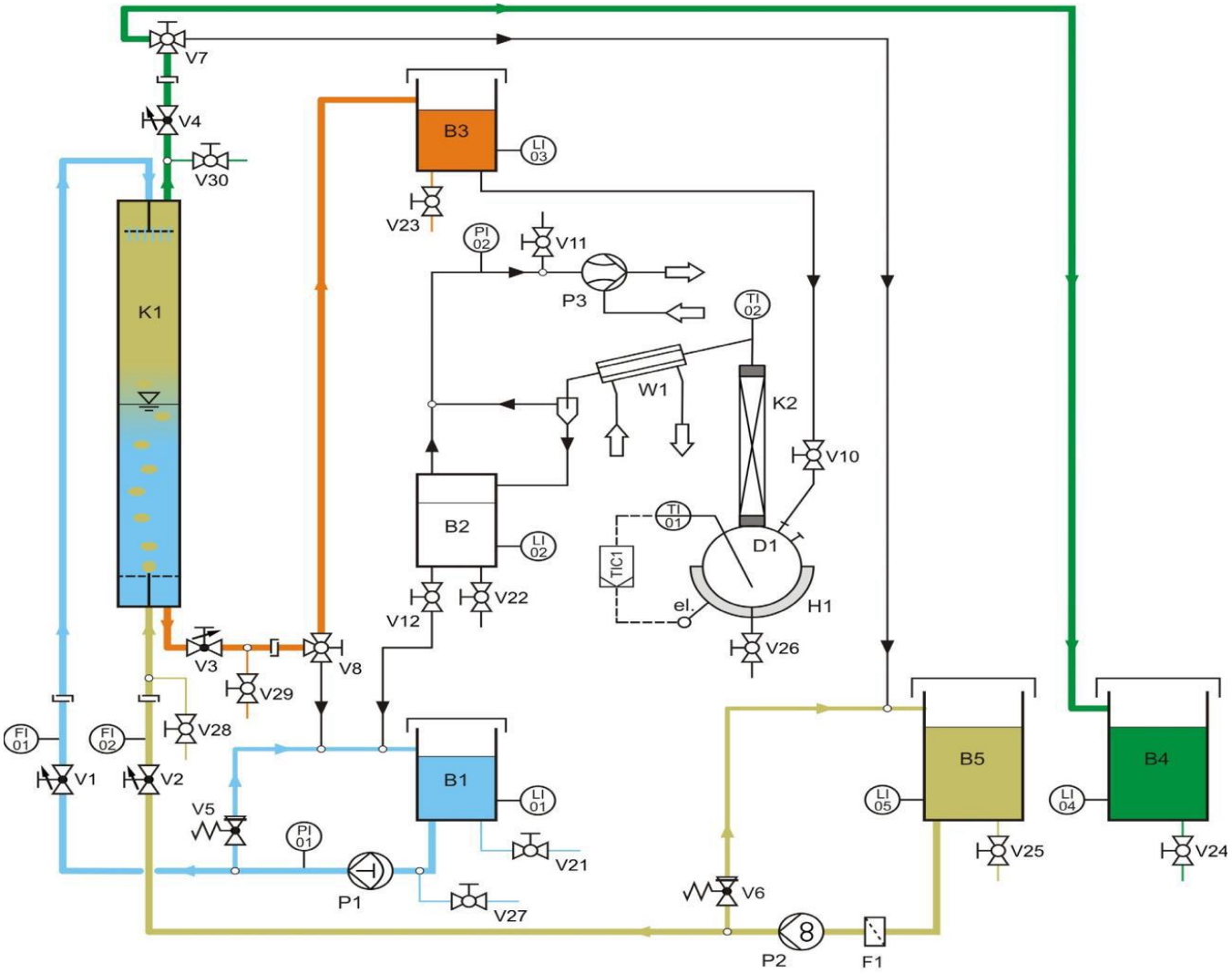



**Fig 6 CE 620 Process diagram, continuous mode variant, without distillation**

**(It shows different feed -product separation with colour coding)**

**11 (d): LLE for continuous column mode operation**

**EXPERIMENTS**

This chapter describes experiments with the recommended **model material system** of **" extraction of ethanol from refined rapeseed oil with water."**

The experiments use:

**Refined rapeseed oil** as carrier liquid,

**Ethanol** as transition component,

**Water** as solvent, as well as methylated spirits, detergent and dilute acetic acid as cleaning agents.

**Table 1 Materials & Cleaning agents used for extraction**

| Materials used | Note |
|---|---|
| Ethanol | Purity > 99,5%. |
| Rapeseed oil | Commercially available. Refined rapeseed oil. |
| Washing-up liquid | Commercially available. Notes such as "high fat dissolving power" are desired. |
| Methylated spirits | Commercially available. Ethanol volume fraction of about 95%. |
| Dilute acetic acid | Commercially available. Vinegar essence, acetic acid about 25%. |

**Experiments with Device- Batch Procedure**

For the material model system, the liquid -liquid system has priority. At the starting of the experiment, by mixing the ethanol and rapeseed oil is used to prepare the feed

The following section describes the **standard experiment** for **liquid-liquid extraction** with **CE 620**.

The **experimental conditions** are:

Extraction of ethanol from rapeseed oil with water.

Mass fraction of ethanol in the feed 10%.

Equal masses of feed and solvent, each 5000g.

Extraction time 180min.

Solvent flow 400 mL/min.

Feed flow 800 mL/min (display value).

## 12) Powder handling unit (Ball mill & V blender)

## 12 (a) V-Blender

Generally, the Ball mills are known as the secondary size reduction equipment. The Ball mill is made in a great many types and sizes and can be used on a greater variety of soft materials than any other type of machine. The feed must be non-abrasive with a hardness of 1.5 or less. A Ball mill consists of a cylindrical shell slowly turning about a horizontal axis and filled to about $1/4^{th}$ of its volume with solid grinding medium (i.e. metallic

balls etc.). When the ball mill is rotated, the grinding elements (balls) are carried to the side of the shell nearly to the top, from where: they fall on the particles under gravity. In a ball mill most of the size reduction is done by impact. The energy expanded in lifting the grinding units are utilizes in reducing the size of the particles. Ball mill can accept a feed size of 12mm or less and deliver a product size in the range of 50μm. The speed of ball mill varies between 60-70 rpm. As the product size becomes fine, the capacity of a mill reduces, the energy requirement increases

The flow and handling characteristics of granular materials are relevant to many process industries, particularly in the handling of powders, pellets, crystals and aggregates. The CEN-MKII introduces students to the behavior of granular materials and is available as three units that can be purchased separately or as a complete set, as follows:

The equipment consists of interchangeable ball mill and Vee blender assemblies that are operated inside a protective enclosure. The enclosure, constructed from solid PVC, incorporates an electric motor with quick release coupling and manually adjustable speed control. A single-piece transparent hinged cover over the top and front of the enclosure allows access to the mill or blender and allows the user to safely observe the operation of the equipment. A safety interlock prevents the motor from operating when the cover is raised.

**12.1 Description of ball mill and V blender- CEN-MKII-11 SOLIDS HANDLING**

The flow and handling characteristics of granular materials are relevant to many process industries, particularly in the handling of powders, pellets, crystals and aggregates. The equipment consists of interchangeable ball mill and Vee blender assemblies that are operated inside a protective enclosure. The enclosure, constructed from solid PVC, incorporates an electric motor with quick release coupling and manually adjustable speed control. A single-piece transparent hinged cover over the top and front of the enclosure allows access to the mill or blender and allows the user to safely observe the operation of the equipment. A safety interlock prevents the motor from operating when the cover is raised.

**12 (a) Ball Mill**

The ball mill is a type of grinder that is used to reduce the size of solid materials using porcelain balls as the grinding medium. The ball mill can also be used to mix different powdered / granular materials. The mill supplied consists of a PVC cylindrical drum that rotates in the horizontal plane. The two ends of the drum are constructed from clear acrylic to aid viewing of the milling operation.


**12 (a): Ball mill in operation**

**12 (b) Vee Blender**

The Vee blender is the gentlest and cost-effective way to blend powdered materials together. The blender supplied consists of a shallow V shaped vessel that is constructed from clear acrylic to aid viewing of the blending / mixing operation.


**12 (b): Description of V-blender**


**12 ©: V blender in operation**

**Theory**

A Ball mill consists of a cylindrical shell slowly turning about a horizontal axis and filled with solid grinding medium (metallic balls, wooden balls or rubber balls). In Ball mill, most of the size reduction is done by impact.

**3. TECHNICAL DETAILS OF V Blender & Ball mill**

**3.1 CEN-MKII-11 Solids Handling**

- Protective enclosure with transparent lid allowing safe operation of a ball mill or Vee blender
- Variable speed ball mill using porcelain balls as the grinding medium. Clear acrylic sides allow visualization of the process
- Variable speed Vee blender constructed from clear acrylic for visualization of the process with dust-tight access cover

**3.2 TECHNICAL SPECIFICATIONS of CEN-MKII-11**

| **Blender** | |
|---|---|
| Speed | Variable from 0 to 50 RPM(Rotations per minute) |
| Total volume | 1.2 liters |
| Working volume | 0.35 liters |

| **Ball mill** | |
|---|---|
| Speed | Variable from 0 to 50 RPM |
| Total volume | 3.5 liters |
| Grinding medium | Porcelain balls – 3.5kg supplied |

**Nomenclature:**

| Nom | Column Heading | Units | Type |
|---|---|---|---|
| $D_{pa}$ | Average feed size | mm | Measured |
| $D_{pb}$ | Average product size | mm | Measured |
| EMC | Energy meter constant | Pulses/kWh | Given |
| G | Acceleration due to gravity | $m/sec^2$ | Given |
| $K_b$ | Bond's constant | Kwh/tons | Calculated |
| m | Feed Rate | Tons/h | Calculated |
| $n_c$ | Critical Speed of Ball Mill | RPM | Calculated |
| $P_1$ | Number of pulses counted at no load condition | * | Measured |
| $P_2$ | Number of pulses counted at loaded condition | * | Measured |
| $P_{act}$ | Actual power required for crushing | kW | Calculated |
| $P_{cal}$ | Calculated Power required for crushing | Kw | Calculated |
| $P_L$ | Power consumed by machine at loaded condition | Kw | Calculated |
| $P_{NL}$ | Power consumed by machine at no load condition | kW | Calculated |
| R | Radius of the Ball Mill | m | Given |
| r | Radius of the Ball | m | Given |
| $t_c$ | Time for Crushing | Sec | Measured |
| $t_{p1}$ | Time for $P_1$ pulses | Sec | Measured |
| $t_{p2}$ | Time for $P_2$ pulses | Sec | Measured |
| $W_f$ | Weight of Feed Taken | Kg | Measured |
| $W_i$ | Work Index of Material | kWh/tons | Given |
| $\eta$ | Crushing Efficiency | % | Calculated |

*Symbols are unitless.